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NEWS 1		Web Page URLs for STN Seminar Schedule - N. America
NEWS 2		"Ask CAS" for self-help around the clock
NEWS 3	OCT 23	The Derwent World Patents Index suite of databases on STN has been enhanced and reloaded
NEWS 4	OCT 30	CHEMLIST enhanced with new search and display field
NEWS 5	NOV 03	JAPIO enhanced with IPC 8 features and functionality
NEWS 6	NOV 10	CA/CAplus F-Term thesaurus enhanced
NEWS 7	NOV 10	STN Express with Discover! free maintenance release Version 8.01c now available
NEWS 8	NOV 20	CA/CAplus to MARPAT accession number crossover limit increased to 50,000
NEWS 9	DEC 01	CAS REGISTRY updated with new ambiguity codes
NEWS 10	DEC 11	CAS REGISTRY chemical nomenclature enhanced
NEWS 11	DEC 14	WPIDS/WPINDEX/WPIX manual codes updated
NEWS 12	DEC 14	GBFULL and FRFULL enhanced with IPC 8 features and functionality
NEWS 13	DEC 18	CA/CAplus pre-1967 chemical substance index entries enhanced with preparation role
NEWS 14	DEC 18	CA/CAplus patent kind codes updated
NEWS 15	DEC 18	MARPAT to CA/CAplus accession number crossover limit increased to 50,000
NEWS 16	DEC 18	MEDLINE updated in preparation for 2007 reload
NEWS 17	DEC 27	CA/CAplus enhanced with more pre-1907 records
NEWS 18	JAN 08	CHEMLIST enhanced with New Zealand Inventory of Chemicals
NEWS 19	JAN 16	CA/CAplus Company Name Thesaurus enhanced and reloaded
NEWS 20	JAN 16	IPC version 2007.01 thesaurus available on STN
NEWS 21	JAN 16	WPIDS/WPINDEX/WPIX enhanced with IPC 8 reclassification data
NEWS 22	JAN 22	CA/CAplus updated with revised CAS roles
NEWS 23	JAN 22	CA/CAplus enhanced with patent applications from India
NEWS 24	JAN 29	PHAR reloaded with new search and display fields
NEWS 25	JAN 29	CAS Registry Number crossover limit increased to 300,000 in multiple databases

NEWS EXPRESS	NOVEMBER 10 CURRENT WINDOWS VERSION IS V8.01c, CURRENT MACINTOSH VERSION IS V6.0c(ENG) AND V6.0Jc(JP), AND CURRENT DISCOVER FILE IS DATED 25 SEPTEMBER 2006.
NEWS HOURS	STN Operating Hours Plus Help Desk Availability
NEWS LOGIN	Welcome Banner and News Items
NEWS IPC8	For general information regarding STN implementation of IPC 8
NEWS X25	X.25 communication option no longer available

Enter NEWS followed by the item number or name to see news on that specific topic.

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FILE 'HOME' ENTERED AT 10:23:38 ON 01 FEB 2007

=> file ca
COST IN U.S. DOLLARS SINCE FILE ENTRY TOTAL SESSION
FULL ESTIMATED COST 0.21 0.21

FILE 'CA' ENTERED AT 10:23:47 ON 01 FEB 2007
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FILE COVERS 1907 - 25 Jan 2007 VOL 146 ISS 6
FILE LAST UPDATED: 25 Jan 2007 (20070125/ED)

New CAS Information Use Policies, enter HELP USAGETERMS for details.

This file contains CAS Registry Numbers for easy and accurate substance identification.

```
=> s silica gel
        515517 SILICA
        488363 GEL
L1       89869 SILICA GEL
                (SILICA(W)GEL)

=> s l1 and liquid chromatograph?
        682656 LIQUID
        405792 CHROMATOGRAPH?
         87558 LIQUID CHROMATOGRAPH?
                (LIQUID(W)CHROMATOGRAPH?)
L2        4609 L1 AND LIQUID CHROMATOGRAPH?

=> s uncoated and l2
         15952 UNCOATED
L3           2 UNCOATED AND L2

=> d ibib abs kwic
```

L3 ANSWER 1 OF 2 CA COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 90:199722 CA
TITLE: Improvements in liquid chromatography column life and method flexibility by saturating the mobile phase with silica
AUTHOR(S): Atwood, J. G.; Schmidt, G. J.; Slavin, W.
CORPORATE SOURCE: Perkin-Elmer Corp., Norwalk, CT, USA
SOURCE: Journal of Chromatography (1979), 171, 109-15
CODEN: JOCRAM; ISSN: 0021-9673
DOCUMENT TYPE: Journal
LANGUAGE: English
AB Liquid chromatog. column life was prolonged by equilibrating the mobile phase with silica using an appropriate column mounted in the oven ahead of the anal. column. An atomic absorption method was adapted for the detection of Si eluting from the column to measure quant. the loss of silica for different anal. conditions. An injection procedure which permitted the atomic absorption burner to take up solution at its optimum rate was used while the liquid chromatog. was used at any lower mobile phase flow-rate. Tricyclic antidepressants were determined with a mobile phase of 40% acetonitrile in H2O at pH 10.7 on uncoated 5-μm silica particles.
TI Improvements in liquid chromatography column life and method flexibility by saturating the mobile phase with silica
AB Liquid chromatog. column life was prolonged by equilibrating the mobile phase with silica using an appropriate column mounted in the oven ahead of the anal. column. An atomic absorption method was adapted for the detection of Si eluting from the column to measure quant. the loss of silica for different anal. conditions. An injection procedure which permitted the atomic absorption burner to take up solution at its optimum rate was used while the liquid chromatog. was used at any lower mobile phase flow-rate. Tricyclic antidepressants were determined with a mobile phase of 40% acetonitrile in H2O at pH 10.7 on uncoated 5-μm silica particles.
IT Silica gel, uses and miscellaneous
RL: USES (Uses)
(in liquid chromatog., mobile phase saturation with)

=> d 2 ibib abs kwic

L3 ANSWER 2 OF 2 CA COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 89:12236 CA
TITLE: Determination of ethinylestradiol in single tablets and its separation from other steroids by high-performance liquid chromatography
AUTHOR(S): Bagon, Kay R.; Hammond, E. W.
CORPORATE SOURCE: Dep. Ind., Lab. Gov. Chem., London, UK
SOURCE: Analyst (Cambridge, United Kingdom) (1978), 103(1223), 156-61
CODEN: ANALAO; ISSN: 0003-2654
DOCUMENT TYPE: Journal
LANGUAGE: English
AB Ethinylestradiol (I) [57-63-6] was rapidly separated from structurally related steroids using a reversed-phase high-performance liquid chromatog. using microfined silica-gel particles chemical bonded with octadecylsilane. Using this method I was directly assayed down to a limit of 10 μg in uncoated tablets using a UV detector set at about 212 nm and in sugar-coated tablets after preextn. with Et2O. The method was sufficiently sensitive to detect I when present with other

steroids as a contaminant.
TI Determination of ethinylestradiol in single tablets and its separation from other steroids by high-performance liquid chromatography
AB Ethinylestradiol (I) [57-63-6] was rapidly separated from structurally related steroids using a reversed-phase high-performance liquid chromatog. using microfined silica-gel particles chemical bonded with octadecylsilane. Using this method I was directly assayed down to a limit of 10 μg in uncoated tablets using a UV detector set at about 212 nm and in sugar-coated tablets after preextn. with Et2O. The method was sufficiently sensitive to detect I when present with other steroids as a contaminant.

```
=> s review/ti
L4        59244 REVIEW/TI

=> s uncoated silica gel
          15952 UNCOATED
         515517 SILICA
         488363 GEL
L5            3 UNCOATED SILICA GEL
                (UNCOATED(W)SILICA(W)GEL)

=> coated silica gel
COATED IS NOT A RECOGNIZED COMMAND
The previous command name entered was not recognized by the system.
For a list of commands available to you in the current file, enter
"HELP COMMANDS" at an arrow prompt (=>).

=> s coated silica gel
         491763 COATED
         515517 SILICA
         488363 GEL
L6          395 COATED SILICA GEL
                (COATED(W)SILICA(W)GEL)

=> s l4 and l5 and l6
L7            0 L4 AND L5 AND L6

=> s separat? epimer?
         329427 SEPARAT?
          21294 EPIMER?
L8            1 SEPARAT? EPIMER?
                (SEPARAT?(W)EPIMER?)

=> d kwic

L8   ANSWER 1 OF 1  CA  COPYRIGHT 2007 ACS on STN
IT   Epimerization
     Resolution (separation)
        (epimerization and resolution of naproxen derivs.)


=> s liquid chromatogra? and epimer?
         682656 LIQUID
         415809 CHROMATOGRA?
          87645 LIQUID CHROMATOGRA?
                (LIQUID(W)CHROMATOGRA?)
          21294 EPIMER?
```

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L9          295 LIQUID CHROMATOGRA? AND EPIMER?

=> s silica gel and l9
         515517 SILICA
         488363 GEL
          89869 SILICA GEL
                  (SILICA(W)GEL)
L10          23 SILICA GEL AND L9

=> s uncoated and l10
          15952 UNCOATED
L11           0 UNCOATED AND L10

=> d l10 ibib abs 1-23

L10  ANSWER 1 OF 23  CA  COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER:        143:153556  CA
TITLE:                   Confirmation of the Structure of (3S)-3-
                         Hydroxyquinine: Synthesis and X-ray Crystal Structure
                         of Its 9-Aceto Analogue
AUTHOR(S):               Sarma, P. V. V. Srirama; Han, Dongmei; Deschamps,
                         Jefferey R.; Cook, James M.
CORPORATE SOURCE:        Department of Chemistry, University of
                         Wisconsin-Milwaukee, Milwaukee, WI, 53201, USA
SOURCE:                  Journal of Natural Products (2005), 68(6), 942-944
                         CODEN: JNPRDF; ISSN: 0163-3864
PUBLISHER:               American Chemical Society
DOCUMENT TYPE:           Journal
LANGUAGE:                English
OTHER SOURCE(S):         CASREACT 143:153556
GI
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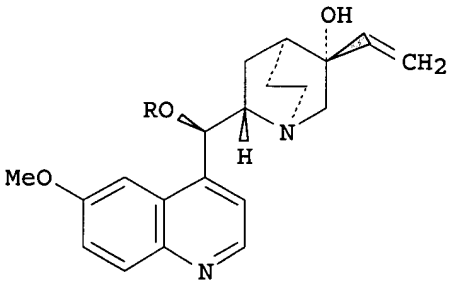


I

AB 3(S)-3-Hydroxyquinine [I; R = H (II)] has been separated from its epimeric mixture at C-3 by conversion into the 9-aceto analog I [R = OAc (III)] followed by chromatog. The mol. structure of III was determined through single-crystal X-ray anal., and this confirms the structure of II, the major metabolite of quinine.

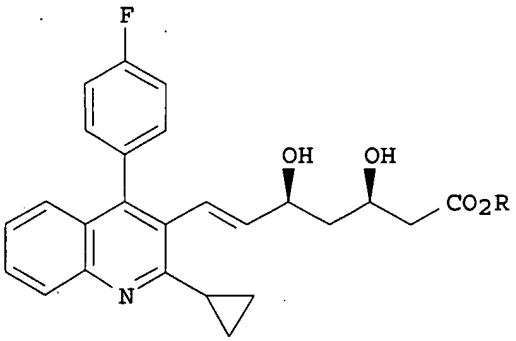
```
REFERENCE COUNT:         17    THERE ARE 17 CITED REFERENCES AVAILABLE FOR THIS
                               RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L10  ANSWER 2 OF 23  CA  COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER:        140:287282  CA
TITLE:                   Purification of a 3,5-dihydroxy-6-heptenoate isomer
INVENTOR(S):             Yoshimura, Yuji; Yasukawa, Masami; Morikiyo, Syuji;
```

Matsumoto, Hiroo; Takada, Yasutaka; Adachi, Michiaki
PATENT ASSIGNEE(S): Nissan Chemical Industries, Ltd., Japan
SOURCE: PCT Int. Appl., 29 pp.
CODEN: PIXXD2
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2004026838	A1	20040401	WO 2003-JP11643	20030911
W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW				
RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG				
CA 2499335	A1	20040401	CA 2003-2499335	20030911
AU 2003260963	A1	20040408	AU 2003-260963	20030911
EP 1539698	A1	20050615	EP 2003-797579	20030911
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR, BG, CZ, EE, HU, SK				
CN 1681790	A	20051012	CN 2003-822333	20030911
JP 2006500405	T	20060105	JP 2004-537553	20030911
US 2006167260	A1	20060727	US 2005-528179	20050317
IN 2005KN00588	A	20060303	IN 2005-KN588	20050406
PRIORITY APPLN. INFO.:			JP 2002-275015 A	20020920
			WO 2003-JP11643 W	20030911

OTHER SOURCE(S): MARPAT 140:287282
GI



I

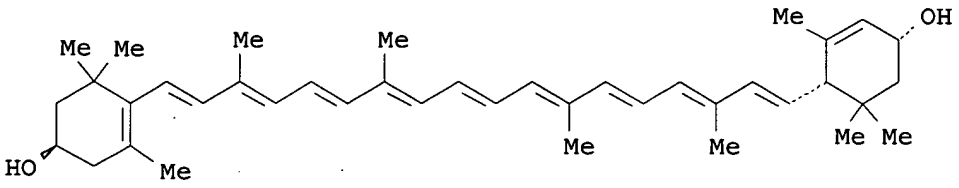
AB An alkyl (3R,5S)-7-[2-cyclopropyl-4-(4-fluorophenyl)quinolin-3-yl]-3,5-dihydroxy-6-heptenoate I [R = alkyl], which is an intermediate for a cholesterol-reducing agent (a HMG-CoA reductase inhibitor), is purified by liquid chromatog. on silica gel.
REFERENCE COUNT: 3 THERE ARE 3 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L10 ANSWER 3 OF 23 CA COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 137:93874 CA
TITLE: Process for obtaining 3'-epilutein via epimerization and column chromatography
INVENTOR(S): Eugster, Conrad Hans; Montoya-Olvera, Ricardo; Torres-Quiroga, Jose-Odon
PATENT ASSIGNEE(S): Industrial Organica, S.A. De C.V., Mex.
SOURCE: U.S., 7 pp.
CODEN: USXXAM
DOCUMENT TYPE: Patent
LANGUAGE: English
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 6420614	B1	20020716	US 2000-685321	20001010
WO 2002030769	A9	20021031	WO 2001-MX73	20011010

W: AE, AG, AL, AU, BA, BB, BG, BR, BZ, CA, CN, CO, CR, CU, CZ, DM, DZ, EC, EE, GD, GE, HR, HU, ID, IL, IN, IS, JP, KE, KP, KR, LC, LK, LR, LT, LV, MA, MG, MK, MN, MX, NO, NZ, PL, RO, SG, SI, SK, TT, UA, UZ, VN, YU, ZA
RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG

PRIORITY APPLN. INFO.: US 2000-685321 A 20001010
OTHER SOURCE(S): CASREACT 137:93874
GI



I

AB A process for obtaining 3'-epilutein (I), by epimerization of a lutein-containing extract with an aqueous solution of a strong organic acid which is slowly added under agitation at room temperature, in the presence of an organic aprotic media, to obtain I (crystals) in a solution which is neutralized with an alkali and extracting the I from said solution by means of an organic media, then washing and drying the crystals and purifying them by chromatog. by means of a chromatog. column. Thus, I was prepared from an enriched lutein (39%) solution via reaction with aqueous 1N. H2SO4 in THF for 14 h at room temperature, neutralizing with NH4OH, partitioning with CH2Cl2, then washing the CH2Cl2 layer with H2O and aqueous NaCl, then drying the organic layer over Na2SO4; the crystals are purified via chromatog. over silica gel.
I can be converted to (3R,3'R)-zeaxanthin via reaction with a strongly alkaline aqueous solution

REFERENCE COUNT: 1 THERE ARE 1 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L10 ANSWER 4 OF 23 CA COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 129:197349 CA
TITLE: High performance liquid chromatography on calixarene-bonded silica gels. II. Separations of regio- and stereoisomers on p-tert-butylcalix[n]arene phases
AUTHOR(S): Gebauer, Sabine; Friebe, Sieglinde; Gubitz, Gerald; Krauss, Gerd-Joachim
CORPORATE SOURCE: Dep. Biochem./Biotechnol., Martin-Luther-Univ. Halle, Halle/S., D-06099, Germany
SOURCE: Journal of Chromatographic Science (1998), 36(8), 383-387
CODEN: JCHSBZ; ISSN: 0021-9665
PUBLISHER: Preston Publications
DOCUMENT TYPE: Journal
LANGUAGE: English
AB The chromatog. behavior of new calix[n]arene-bonded (n = 4, 6, 8) silica gels are described. Cavities of different size and shape are formed depending on the number of aromatic moieties. The differences in ring size were used to study chromatog. selectivities towards analytes of various substance classes, including disubstituted aroms., uracil derivs., and estradiol epimers. The authors' results indicate that these calixarene-bonded phases show a high resolution power for regio- and stereoisomers.
REFERENCE COUNT: 21 THERE ARE 21 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L10 ANSWER 5 OF 23 CA COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 119:176880 CA
TITLE: Preparation and determination of zinc(II) chlorophylls by reversed-phase high-performance liquid chromatography
AUTHOR(S): Inoue, Hidenari; Imai, Miki; Naemura, Takashi; Furuya, Kenji; Shizuri, Yoshikazu
CORPORATE SOURCE: Faculty of Science and Technology, Keio University, 3-14-1 Hiyoshi, Kohoku-ku, Yokohama, 223, Japan
SOURCE: Journal of Chromatography (1993), 645(2), 259-64
CODEN: JOCRAM; ISSN: 0021-9673
DOCUMENT TYPE: Journal
LANGUAGE: English
AB A preparative method for zinc(II) chlorophyll a and b [Zn(II)-chl-a and -b] was developed on the basis of their purification by semi-preparative high-performance liquid chromatog. Zn(II)-chl-a and -b and the corresponding epimers were separated and determined with an ODS (C18 chemical bonded silica gel) column using methanol-acetone (75:25, volume/volume) as mobile phase. Linear calibration graphs were obtained over the concentration range 0-50 μg cm-3 of each zinc(II) chlorophyll with photometric detection at 425 nm. The present HPLC determination provides an accurate and conventional method with a detection limit of 3.5 ng cm-3 for Zn(II)-chl-a, 2.5 ng cm-3 for Zn(II)-chl-a' and 3.0 ng cm-3 for Zn(II)-chl-b with a relative standard deviation of less than 2.3% (n = 10). The anal. values obtained for synthetic samples by a spectrophotometric method were confirmed to be high compared with those determined by the proposed HPLC method.

L10 ANSWER 6 OF 23 CA COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 119:117615 CA
TITLE: Method for separation of 24-epimers of 24-hydroxycholesterol derivatives by liquid chromatography
INVENTOR(S): Saito, Yoichi; Yarino, Tatsuo; Fujii, Takao
PATENT ASSIGNEE(S): Teijin Ltd, Japan
SOURCE: Jpn. Kokai Tokkyo Koho, 7 pp.
CODEN: JKXXAF
DOCUMENT TYPE: Patent
LANGUAGE: Japanese
FAMILY ACC. NUM. COUNT: 1
PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 05043591	A	19930223	JP 1991-246480	19910902
JP 2986592	B2	19991206		

PRIORITY APPLN. INFO.: JP 1990-235797 A1 19900907
OTHER SOURCE(S): MARPAT 119:117615
AB An 24-epimeric mixture of 24-hydroxycholesterol derivs. is separated by liquid chromatog. using as a packing material, a silica gel modified with a Si compound R1SiR2R3R4 (R1 = C1-22 aliphatic or C6-10 arom. group; R2 - R4 = halo, lower alkoxy, lower alkyl, where at least one of R2 - R4 = halo or lower alkoxy). The packing material shows Kp = -1 to 0.2 in an elution liquid system of MeCN/H2O (30/70), wherein Kp = [(pyridine retention time) - (phenol retention time)]/(pyridine retention time). Preferably the hydroxycholesterol derivs. separated are 1α,24-dihydroxycholesterol (I) and 1α,24-dihydroxycholesta-5,7-diene (II). Thus, 20% THF solution of a mixture of (1α,24R)- and (1α,24S)-I was fed to a HPLC column YMC-A-802 packed with C4-120-S5, a silica gel modified with BuSiR2R3R4 (at least one of R2 - R4 = Me or Cl), using MeCN/H2O (6/4) as a mobile phase at room temperature to give (1α,24S)-I with separation degree of 1.2, wherein the separation degree = [(R-isomer retention time) - (S-isomer retention time)]/[[(R-isomer half width) + (S isomer half width)]/(chart speed)]. Addnl. used were YMC-A-202, -302, -402, and YMC-SH-343-50AQ column. II epimers were separated by a column packed with ODS-50AQ.

L10 ANSWER 7 OF 23 CA COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 117:229369 CA
TITLE: Quantitative derivatization and high-performance liquid chromatographic analysis of cyanobacterial heterocyst-type glycolipids
AUTHOR(S): Davey, Mark W.; Lambein, Fernand
CORPORATE SOURCE: Lab. Fysiol. Scheikd., Rijksuniv. Gent, Ghent, B-9000, Belg.
SOURCE: Analytical Biochemistry (1992), 206(2), 323-7
CODEN: ANBCA2; ISSN: 0003-2697
DOCUMENT TYPE: Journal
LANGUAGE: English
AB Procedures are described for the rapid and quant. anal. of cyanobacterial heterocyst-type glycolipids (HGs) by normal-phase HPLC of their per-O-benzoylated derivs. Total lipids are obtained from 1 mL of nitrogen-fixing cyanobacterial culture by triplicate extraction with chloroform/methanol, 1/1 (volume/volume), and the HGs are isolated from other complex lipids by preparative silica gel TLC. A C18 solid-phase extraction cartridge is used to ensure quant. salt-free recovery of the HGs, and the purified glycolipids are then rendered uv-absorbing by a per-O-benzoylation derivatization reaction for which optimal conditions

have been established. Derivs. are analyzed within 12 min on a 3-μm silica HPLC column using a linear gradient of 2-propanol in n-hexane and uv monitoring at 230 nm. The reaction product was also used to determine the relative proportions of the glycosyl and galactosyl epimers of individual members of this class of glycolipid.

L10 ANSWER 8 OF 23 CA COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 116:28266 CA
TITLE: Isolation of doxycycline, 6-epidoxycycline and 2-acetyl-2-decarboxamidometacycline from commercial metacycline by preparative column liquid chromatography on silica gel
AUTHOR(S): Naidong, Weng; Verresen, K.; Busson, R.; Roets, E.; Hoogmartens, J.
CORPORATE SOURCE: Inst. Farm. Wetensch., Kathol. Univ. Leuven, Louvain, B-3000, Belg.
SOURCE: Journal of Chromatography (1991), 586(1), 67-72
CODEN: JOCRAM; ISSN: 0021-9673
DOCUMENT TYPE: Journal
LANGUAGE: English
AB Isolation of doxycycline, 6-epidoxycycline and 2-acetyl-2-decarboxamidometacycline from com. metacycline was achieved by preparative column liquid chromatog. on silica gel, previously impregnated with EDTA. Careful control of the pH of EDTA allowed fine tuning of the separation The mobile phases were composed of dichloromethane, methanol and 0.1 mM EDTA at pH 9.0 or 6.0. Structures were confirmed with NMR spectroscopy. The presence of doxycycline and its 6-epimer in com. metacycline has not previously been described. The presence of the 2-acetyl derivative was not surprising since analogs 2-acetyl derivs. have been identified in other tetracyclines.

L10 ANSWER 9 OF 23 CA COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 108:10995 CA
TITLE: Separation of non-polar sesquiterpene olefins from tolu balsam by high-performance liquid chromatography; silver perchlorate impregnation of a prepacked preparative silica gel column
AUTHOR(S): Friedel, Horst Dieter; Matusch, Rudolf
CORPORATE SOURCE: Inst. Pharm. Chem. Lebensmittelchem., Philipps-Univ. Marburg, Marburg, 3550, Fed. Rep. Ger.
SOURCE: Journal of Chromatography (1987), 407, 343-8
CODEN: JOCRAM; ISSN: 0021-9673
DOCUMENT TYPE: Journal
LANGUAGE: English
AB A separation of nonpolar sesquiterpenoid olefins in EtOH exts. of tolu balsam was achieved by using a AgClO4-loaded HPLC column (LiChrosorb Si 60) and pentane-Et2CO (80:20 and 99:1) as the mobile phase. The method showed high efficiency and good reproducibility, especially in the separation of epimeric compds. which have similar spectroscopic properties.

L10 ANSWER 10 OF 23 CA COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 105:205623 CA
TITLE: Separation of allo bile acid stereoisomers by thin-layer and high-performance liquid chromatography
AUTHOR(S): Iida, Takashi; Momose, Toshiaki; Shinohara, Toshiyuki; Goto, Junich; Nambara, Toshio; Chang, Frederic C.
CORPORATE SOURCE: Coll. Eng., Nihon Univ., Koriyama, 963, Japan
SOURCE: Journal of Chromatography (1986), 366, 396-402

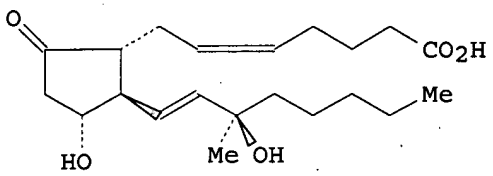
11-dehydrocorticosterone, pregnenolone, and testosterone were described and all compds. were separated in a 90% yield. The extrapolation of the conditions used for TLC to preparative HPLC was discussed.

L10 ANSWER 13 OF 23 CA COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 102:39210 CA
TITLE: Liquid chromatographic separation of diastereomers and structural isomers on cyclodextrin-bonded phases
AUTHOR(S): Armstrong, Daniel W.; DeMond, Wade; Alak, Ala; Hinze, Willie L.; Riehl, Terrence E.; Bui, Khanh H.
CORPORATE SOURCE: Dep. Chem., Texas Tech. Univ., Lubbock, TX, 79409, USA
SOURCE: Analytical Chemistry (1985), 57(1), 234-7
CODEN: ANCHAM; ISSN: 0003-2700
DOCUMENT TYPE: Journal
LANGUAGE: English
AB Compds. (80) were separated from their isomers by liquid chromatog. on cyclodextrin-bonded columns. A variety of structural isomers (including polycyclic aromatic hydrocarbons and prostaglandins), geometric isomers, and steroid epimers were examined Cyclodextrin-bonded packings appear to be more widely applicable than either normal or reversed-phase packings for these types of sepns. Indeed, compds. that cannot be well resolved on more traditional columns are often easily separated on this stationary phase. The separation mechanism is based on inclusion complex formation and is responsible for the unusual but often predictable selectivities observed

L10 ANSWER 14 OF 23 CA COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 100:31672 CA
TITLE: Preparation of chlorophylls and pheophytins by isocratic liquid chromatography
AUTHOR(S): Watanabe, Tadashi; Hongu, Akinori; Honda, Kenichi; Nakazato, Masataka; Konno, Mitsuo; Saitoh, Sadao
CORPORATE SOURCE: Fac. Eng., Univ. Tokyo, Tokyo, 113, Japan
SOURCE: Analytical Chemistry (1984), 56(2), 251-6
CODEN: ANCHAM; ISSN: 0003-2700
DOCUMENT TYPE: Journal
LANGUAGE: English
AB Isocratic high-performance liquid chromatog. (HPLC) with silica gel as a stationary phase provides a powerful means for rapid preparative isolation (on a 20-50 mg level) of chlorophylls (a, a', b, and b') and pheophytins (a, a', b, and b'). The purity and identical of the isolated pigments were confirmed by complete elemental analyses and anal. HPLC; the purity levels were 99.9, 99.5, 99.5, 99.4, 95, 91, 99.5, and 85% for chlorophyll a, a', b, and b' and pheophytin a, a', b, b', resp., with the sole impurities being almost totally corresponding epimers. UV-visible spectrometric data (in Et2O, Me2CO, and C6H6) and CD spectra (in C6H6) of the purified pigments are presented.

L10 ANSWER 15 OF 23 CA COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 99:110835 CA
TITLE: Liquid chromatographic assay of arbaprostil
AUTHOR(S): Peng, G. W.; Sood, V. K.
CORPORATE SOURCE: Pharm. Res. Dev., Upjohn Co., Kalamazoo, MI, 49001, USA
SOURCE: Journal of Liquid Chromatography (1983), 6(8), 1499-511
CODEN: JLCHD8; ISSN: 0148-3919
DOCUMENT TYPE: Journal
LANGUAGE: English

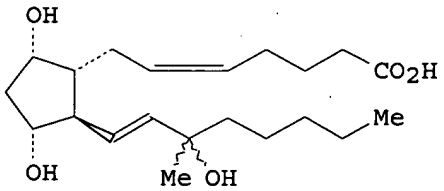
GI



I

AB Arbaprostil (I) [55028-70-1] was extracted from liquid formulations with Et2O-CHCl3, the exts. were evaporated, and I was derivatized with p-nitrophenacyl bromide in the presence of N,N-diisopropylethylamine. The samples were chromatographed on a silica gel column with MeCN-CH2Cl2-H2O (150:350:2.5) mobile phase. The p-nitrophenacyl esters, the 15-S epimer [35700-27-7], and the degradation products of I were separated When monitored by UV absorption, the degradation products could not be detected as they eluted near the solvent front under the peak of the derivatization reagent. The chromatog. responses were linear with the concns. of I.

L10 ANSWER 16 OF 23 CA COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 97:203288 CA
TITLE: Determination of 15-methylprostaglandin F2α by derivatization high-pressure liquid chromatography
AUTHOR(S): Wang, Zhongshan; Zhu, Yaohua; Zhang, Shuliang; Zhu, Jiyu; Wang, Meili
CORPORATE SOURCE: Shanghai Inst. Drug Control, Shanghai, Peop. Rep. China
SOURCE: Yaoxue Xuebao (1982), 17(8), 603-8
CODEN: YHHPAL; ISSN: 0513-4870
DOCUMENT TYPE: Journal
LANGUAGE: Chinese
GI

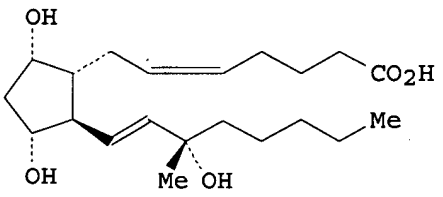


I, ?-OH, ?-Me
II, ?-OH,?-Me

AB After conversion to their p-bromophenacyl esters at room temperature, epimeric 15-(R)- (I) [35864-81-4] and 15-(S)-methylprostaglandin F2α (II) [35700-23-3] were completely separated by HPLC on a microparticulate silica gel column using CHCl3-EtOAc (1:1) as the eluent. The derivs. were detected by UV at 257 nm. The ratio of the epimers in a number of samples were accurately determined by this method. Several samples were quant. determined using pure 15-(S)-PGF2α p-bromophenacyl ester (m 85.apprx.86°) as the external standard which was prepared Other prostaglandins including PGF2α

[551-11-1], PGE1 [745-65-3], PGE2 [363-24-6] and ω-ethyl-13-dehydro-PGF2α [36950-85-3] were also determined by the same method and their relative retention time compared to 15-(S)-PGF2α p-bromophenacyl ester were given. The method proposed was suitable for accurate determination of I and II and their relative contents in samples.

L10 ANSWER 17 OF 23 CA COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 94:109418 CA
TITLE: Comparison of two high-pressure liquid chromatographic assays for carboprost, a synthetic prostaglandin
AUTHOR(S): Brown, Leo W.; Carpenter, Bruce E.
CORPORATE SOURCE: Control Res. Dev., Upjohn Co., Kalamazoo, MI, 49002, USA
SOURCE: Journal of Pharmaceutical Sciences (1980), 69(12), 1396-9
CODEN: JPMSAE; ISSN: 0022-3549
DOCUMENT TYPE: Journal
LANGUAGE: English
GI

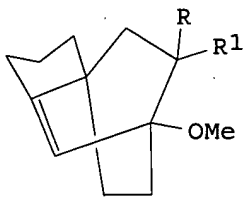


I

AB A high-pressure liquid chromatog. assay for carboprost tromethamine (I tromethamine) [58551-69-2] as the bulk drug and in a sterile solution formulation is described. The procedure involves derivatization of the prostaglandin to form the UV-absorbing naphthacyl ester, which then is chromatographed on a silica gel column using CH2Cl2-1,3-butanediol-H2O (496:4:0.25) as the mobile phase. This procedure is compared with a nonderivatization procedure with refractive index detection. Both procedures sep. the 15R-epimerof I [35864-81-4] from I, but only the derivatization procedure seps. the 5-trans-isomer of I [76498-29-8]. Possible reasons for the better separation using the derivatization procedure are discussed. Both procedures gave a coefficient of variation of .apprx.1% for I. The derivatization procedure gave a coefficient of variation of .apprx.7% for the 15R-epimer and 5--trans-isomer when present at 2% of the I level.

L10 ANSWER 18 OF 23 CA COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 93:185599 CA
TITLE: Liquid chromatographic resolution of epimeric Diels-Alder adducts on silica gel using binary solvent systems
AUTHOR(S): Hara, Shoji; Ohkuma, Toshikazu; Nagaoka, Hiroto; Yamada, Yasuji
CORPORATE SOURCE: Tokyo Coll. Pharm., Tokyo, 192-03, Japan
SOURCE: HRC & CC, Journal of High Resolution Chromatography and Chromatography Communications (1980), 3(4), 193-4
CODEN: HCJCDB; ISSN: 0344-7138
DOCUMENT TYPE: Journal

LANGUAGE: English
GI



I

AB Resolution of I (R = Cl R1 = CN; R = CN, R1 = Cl) was accomplished on a silica gel column using a hexane-2-propanol system.

L10 ANSWER 19 OF 23 CA COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 89:175853 CA
TITLE: Separation of peracetylated mono- and disaccharides and quantitative analysis of guaran by high-performance liquid chromatography on silica gel
AUTHOR(S): Thiem, Joachim; Schwentner, Jens; Karl, Horst; Sievers, Axel; Reimer, Joachim
CORPORATE SOURCE: Inst. Org. Chem. Biochem., Univ. Hamburg, Hamburg, Fed. Rep. Ger.
SOURCE: Journal of Chromatography (1978), 155(1), 107-18
CODEN: JOCRAM; ISSN: 0021-9673
DOCUMENT TYPE: Journal
LANGUAGE: English
AB High-performance liquid chromatog. on silica gel was successfully applied to the separation of 15 monosaccharide peracetates. The solvent system Me2CO-n-hexane gave the most efficient sepns. at the optimal flow-rate. Dependences of retention times or capacity factors on flow-rate or pressure were determined for all derivs. The chromatograms obtained showed clear sepns. of all derivs., including epimers and anomers. Similarly, 12 disaccharide peracetates were analyzed. The best solvent system was Me2CO-pentane, and again the resulting chromatograms showed excellent sepns. For detection both a UV photometer and a refractive index detector could be used, depending on the eluent. Acid cleavage and subsequent peracetylation of the polysaccharide guaran gave a mixture of peracetylated monosaccharides. The interpretation of the chromatogram led to qual. results and, after careful calibration, to quant. results for the ratio of galactose to mannose residues in guaran.

L10 ANSWER 20 OF 23 CA COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 66:28981 CA
TITLE: Dehydrochlorination of 3β-chloro-5β-cholestane. New syntheses of 5β-cholest-2-ene and 5β-cholest-3-ene
AUTHOR(S): Bellucci, Giuseppe; Macchia, F.; Malaguzzi, Valerio
CORPORATE SOURCE: Univ. Pisa, Pisa, Italy
SOURCE: Tetrahedron, Supplement (1966), (41), 4973-8
CODEN: TETSAE; ISSN: 0563-2072
DOCUMENT TYPE: Journal
LANGUAGE: English
AB Elimination reactions in the 3-substituted-5β-cholestanes (I) were investigated by means of gas liquid chromatography at 190° over 2 m. glass columns containing 1% NGS on Chromosorb W.

Quinoline dehydrochlorination of I (R = Cl) (II), m. 122-4°, yielded 5β-cholest-3-ene (III) and 5β-cholest-2-ene (IV) in 55:45 ratio, showing retention times of 1.06, 0.94 relative to 5α-cholestane. Chromatography of the mixture over silica gel-AgNO3 and elution with petr. ether gave pure III. Reduction of 4β-bromo-5β-cholestan-3-one, m. 110-12°, with NaBH4 in absolute alc. at 25° gave a mixture of epimeric bromohydrins, transformed by refluxing with Zn in AcOH to pure III, m. 48-9° (EtOAc-MeOH), [α] 23 D 19.2° (c 1.583, CHCl3); 3α, 4β-dibromo derivative m. 98.5-100.0°. Abnormal opening of 4β, 5-epoxy-5β-cholestan-3-one with H2SO4-Me2CO followed by acetylation and the resulting 2α-acetoxycholest-4-en-3-one hydrogenated over 10% Pd-C in MeOH-dioxane saturated with NaHCO3 gave 65% 2α-acetoxy-5β-cholestan-3-one (V), m. 137-9° (petr. ether), [α] 28 D 0.5°, readily epimerized in AcOH-HBr to the more stable 2β-acetoxy-5β-cholestan-3-one, m. 150.5-2.0° (petr. ether), [α] 27 D -4°. V submitted to the Wolff-Kishner reaction under Huang-Minlon conditions yielded IV together with about 30% 5β-cholestane (VI), m. 70-1°. The mixture brominated at 18° in CHCl3 and heated (N atmospheric) at 180-200° to isomerize the dibromide of II to a more stable form and the product chromatographed on silica gel and eluted with petr. ether gave in succession VI and a dibromide, m. 165-7° (Me2CO), [α] 27 D 71.4° (c 1.060, CHCl3), refluxed in AcOH with Zn to yield pure IV, m. 47.5-8.0° (Me2CO), [α] 28 D 19.9° (c 1.720, CHCl3). IV brominated as above gave a dibromide, m. 74-6° (Me2CO), isomerized to the above-mentioned compound It was surmised that the compds., m. 74-6° and 165-7°, are the diaxial and diequatorial 2α, 3β- and 2β, 3α-dibromo-5β-cholestanes.

L10 ANSWER 21 OF 23 CA COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 64:104458 CA
ORIGINAL REFERENCE NO.: 64:19688f-h,19689a-c
TITLE: Terpenoids. LXXXVII. Structure of nardol
AUTHOR(S): Sastry, S. D.; Maheshwari, M.; Bhattacharyya, S. C.
CORPORATE SOURCE: Natl. Chem. Lab., Poona, India
SOURCE: Tetrahedron Letters (1966), (10), 1035-42
CODEN: TELEAY; ISSN: 0040-4039
DOCUMENT TYPE: Journal
LANGUAGE: English

AB cf. CA 62, 4058f; preceding abstract Isolation from the dark brown variety of Nardostachys jatamansi roots by use of a low temperature solvent extraction procedure gave 4.38% yield of a concentrate sapd. to yield 2.2:97.8 acidic-neutral fractions. The high vacuum distillate from the neutral fraction chromatographed over Al2O3 (activity III) and elution with petr. ether, C6H6, Et2O, and alc. gave 4.5% calarene, 4.5% aristolene, 0.48% valeranone, and several unidentified compds. from the petr. ether fraction. Careful rechromatography of the C6H6 eluate on Al2O3 (activity II) yielded 0.36% of a new alc., nardol (I), C15H26O, b0.5 120-5°, n26D 1.5005, [α]27D -10.17° (c 2.36, CHCl3), ν 1648, 895 cm.-1, λ 220 mμ (ε 560), giving a pos. C(NO2)4 yellow color but containing only 1 double bond (quant. hydrogenation over PtO2 in alc. AcOH). I was stable in Jones reagent and contains a tertiary OH group. I dehydrogenated 13 hrs. with Se yielded Se-guaiazulene (trinitrobenzene adduct m. 148-9°). S dehydrogenation gave S-guaiazulene (trinitrobenzene adduct m. 147-8°). Continued dehydrogenation (48 hrs.) gave a mixture of naphthalenic hydrocarbons, eudalene and cadulene (25% 40:60), showing that I contains the basic azulenic skeleton. The N.M.R. spectrum of I showed a methylenic doublet

at τ 5.45 and the presence of Me2CH and Me groups at τ 9.15, 9.18, and 8.89. The position of the double bond was demonstrated by ozonization to an oxo alc. (II), ν 1712 cm.-1 with a CO group on a 7-membered ring. The N.M.R. spectrum of dihydronardol (III), τ 8.6, 8.95, 9.06, 9.15, 9.17 showed the presence of 1 Me group on a C atom carrying an O function and 3 secondary Me groups. On the basis of the above data structures were assigned to I, II, and III, resp. The position of the OH group at C was confirmed. III benzoate was pyrolyzed and the epimeric hydrocarbon mix., ν 803, 892, 1650 cm.-1 separated by preparative thin layer chromatography on silica gel impregnated with AgNO3 to give a hydrocarbon, N.M.R. γ 4.9, 8.39 [(J = 2 cycles/sec. (cps.)], 9.05, 9.11, 9.14, and its isomer, τ 4.67, 8.19 (J = 2 cps.), 9.13 (J = 6 cps.), indicating that the mixts. may be represented by the given structures (IV or V). Dehydration of the mixture of C10 epimers, III with SOCl2 in C5H5N also gave the epimeric mixture IV, V and the mode of dehydration suggested the axial nature of the OH group. The epimeric mixture of IV, V hydrogenated catalytically gave a mixture of saturated hydrocarbons, [α]27D -31.06°, shown by gas liquid chromatographic analysis to be a mixture of 3 epimers in the ratio 65:25:10. The retention time of the major constituent was identical with that of guaiane, though the possibility of trans ring junctures in the major constituent cannot be ruled out.

L10 ANSWER 22 OF 23 CA COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 63:45835 CA
ORIGINAL REFERENCE NO.: 63:8221b-h,8222a
TITLE: Macrocyclic musk compounds. IX. New synthesis of cyclohexadecenone and cyclohexadecanone from aleuritic acid
AUTHOR(S): Mathur, H. H.; Bhattacharyya, S. C.
CORPORATE SOURCE: Natl. Chem. Lab., Poona, India
SOURCE: Tetrahedron (1965), 21(6), 1537-40
CODEN: TETRAB; ISSN: 0040-4020
DOCUMENT TYPE: Journal
LANGUAGE: English
GI For diagram(s), see printed CA Issue.
AB cf. CA 58, 6709f; 63, 4177f. Aleuritic acid (100 g.) was stirred in pure dry Me2CO containing 2 ml. concentrated H2SO4 and the isopropylidene derivative oxidized with 75 g. KMnO4 (CA 60, 11839h), filtered, and the MnO2 sludge dispersed in 500 ml. H2O and bubbled through with SO2, the oily product deacetonylated by boiling with stirring in 500 ml. H2O containing 25 ml. 5N H2SO4 30 min., the cooled mixture filtered, and the washed and dried product crystallized from alc. gave 68 g. HO2C(CH2)5CH(OH)CH(OH)(CH2)7CO2H (I), m. 129.5-30°, ν 3100, 1680, 1130 cm.-1 I (100 g.) treated with 1.36 l. 15% HBr in AcOH, the crude dibromo derivative (140 g.) esterified, and the diethyl ester (n27D 1.4865) debrominated with Zn dust in alc. gave 86 g. trans-EtO2C(CH2)6CH:CH(CH2)7-CO2Et (II), b0.05 145-6°, n29D 1.4510, ν 969 cm.-1 (trans-CH:CH), converted to trans-hexadec-7-enedioic acid, m. 96-7° (AcOH), ν 2860, 1684, 962 cm.-1 II (20 g.) cyclized with Na in xylene gave 14 g. mixture of acyloins, b0.05 133-8°, ν 3300, 1711, 966 cm-1. The acyloins were acetylated and the mixed acetates (11.6 g., b0.04 131-3°, ν 1732, 1242, 970 cm.-1) reduced with 6 g. Ca in 750 ml. liquid NH3, the product (9.5 g., n29D 1.4980) chromatographed over 190 g. Al2O3 (grade II) and eluted with 100 ml. C6H14 gave a ketone-free fraction (0.5 g.), rechromatographed over 50 g. Al2O3 (grade I), eluted with C6H14, and the eluate distilled over 0.37 g. Na to give material, b1.0 180°, n28D 1.4695, ν 2920, 2646, 1439 1361, 1333, 1290, 1147, 1012, 962, 883, 716 cm.-1, containing some impurity

according to gas-liquid chromatographic analysis. The NMR signal at τ 9.13 indicated the presence of a secondary Me group possibly formed due to ring contraction. Elution with 400 ml. C6H14 gave 0.46 g. material, distilled to yield the 2 cyclohexadecenones (III, 0.37 g.), C16H28, b0.01 170°, n28D 1.4967, ν 2950, 2874, 1709, 1458, 1402, 1372, 1290, 1029, 971 cm.-1; semicarbazone m. 180-1°, ν 3445, 3160, 1692, 1581, 1311, 1294, 1208, 1137, 1104, 1079, 965, 764, 722 cm.-1 Further elution with 100 ml. 1:2 C6H14-C6H6 gave an unidentified fraction (0.79 g.), n29D 1.5041, ν 3505, 1708 cm.-1 Elution with 600 ml. C6H6 yielded 1.93 g. material, n29D 1.5005, ν 3355, 1703 cm.-1, rechromatographed over 100-fold amount Al2O3 and eluted with 1:1 C6H14-C6H6, to give a sticky solid, m. 61-70°, repeatedly sublimed to yield the alcs. (IV), m. 70-2°, ν 3320, 1455, 1347, 1229, 1189, 1077, 1051, 1009, 964, 869, 722 cm.-1 Further elution with 500 ml. Et2O and the fraction (1.86 g.) crystallized from C6H14 yielded trans-cyclohexadec-8-ene-1,2-diol (V), m. 87.0-8.5°, ν 3355, 2355, 1292, 1174, 1133, 1072, 1012, 964 cm.-1 Elution with 500 ml. CHCl3 gave an epimer of V, m. 76-7° (C6H14), ν 3250, 963 cm.-1, whereas the final elution with 350 ml. alc. yielded the other epimer, m. 111-12° (C6H14), ν 3200, 964 cm.-1 II hydrogenated over Raney Ni gave 18.6 g. saturated ester EtO2C(CH2)14CO2Et, m. 37.0-7.5°, cyclized with Na in xylene to give 9.75 g. acyloin, b0.05 139-41°, ν 3400, 1716, 1453, 1410, 1353, 1297, 1060 cm.-1; acetate, b0.005 125-7°, ν 1729, 1440, 1360, 1237, 1114, 1022 cm.-1 The acetate (7.1 g.) reduced with 3.85 g. Ca in 500 ml. liquid NH3 and the solid product (5.64 g.) chromatographed on 100 g. Al2O3 (grade II), eluted with 50 ml. C6H14, and the hydrocarbon, 967 cm.-1, treated with oleum, chromatographed over 60-fold amount of activated silica gel, and eluted with C6H14, the eluate (0.45 g.) rechromatographed over 60 g. Al2O3 and eluted with C6H14 gave 0.36 g. hydrocarbon, distilled over 0.18 g. Na to yield 87% pure C16H32, b0.05 150°, n26D 1.4600, ν 2830, 1458, 1375, 1300, 721 cm.-1, N.M.R. signal at τ 9.12, indicating the presence of a related hydrocarbon containing a secondary Me group. Elution with 450 ml. C6H14 gave 0.43 g. fraction, distilled to yield material, b0.001 158°, λ 234 mμ (ε 3709), apparently containing some α,β-unsatd. ketone. The material hydrogenated and the product crystallized from MeOH gave cyclohexadecanone, m. 62.0-3.5°, λ 1711, 1412, 1284, 1206, 1177, 1149, 1123, 1086, 1046, 730 cm.-1; semicarbazone m. 184.0-4.5°, ν 3445, 3085, 2340, 1660, 1581, 1345, 1231, 1080, 770 cm.-1 Further elution with 900 ml. 1:1 C6H14-C6H6 gave 0.61 g. fraction, crystallized from MeOH and sublimed to yield pure cyclohexadecanol, m. 82.0-3.5°, ν 3310, 2940, 1335, 1274 cm.-1 Elution with 500 ml. Et2O and 200 ml. alc. produced 0.83 g. and 2.14 g. fractions, repeatedly crystallized from C6H14 to give the epimeric cyclohexadecane-1,2-diols, m. 102-3°, and 107-8°, ν 3240, 2350, 1299, 1171, 1149, 1129, 1058, 917 cm.-1 Two intermediate fractions which absorbed in both the hydroxyl and carbonyl regions were not processed further.

L10 ANSWER 23 OF 23 CA COPYRIGHT 2007 ACS on STN

ACCESSION NUMBER:	58:40176 CA
ORIGINAL REFERENCE NO.:	58:6870g-h,6871a-h
TITLE:	Stereochemistry of occidentalol and its hydrogenation products
AUTHOR(S):	v. Rudloff, E.; Erdtman, H.
CORPORATE SOURCE:	Natl. Regional Res. Lab., Saskatoon, Can.
SOURCE:	Tetrahedron (1962), 18, 1315-29 CODEN: TETRAB; ISSN: 0040-4020
DOCUMENT TYPE:	Journal

LANGUAGE: Unavailable
GI For diagram(s), see printed CA Issue.
AB cf. CA 55, 23938e. The milled heartwood of a mature Eastern white cedar (Thuja occidentalis) extracted with Me2CO and the dark viscous product poured into Et2O, the decanted Et2O extracted 3 times with 10% KOH and the washed and dried extract evaporated, the neutral residue fractionally distilled through a spinning band column at 18 mm. and the fraction, b78 148-9°, purified by crystallization from petr. ether and by sublimation gave occidentalol (I), m. 97.5-8.0°, [α]25D 363.2° (c 1.6, CHCl3). I was analyzed by gas-liquid chromatography by using coiled C7 tubing (6 ft. + 0.25 in.) containing adipate polyethylene glycol polyester, polyphenyl ether, and SE-30 silicone polymer on Chromosorb W in the ratio of 1:6 and was better than 99-5% pure. The relative retention time (RRT) with respect to cedrol on adipate polyester at 160° in 60 ml. He/min. was 0.92. I (0.5 g.) in CH2Cl2 at -70° ozonized and the blue solution extracted with H2O, the extract treated with excess PhNHNH2.HCl and the phenylhydrazones chromatographed from C6H6 on silica gel, eluted with C6H6 and the fraction crystallized from C6H6-petr. ether gave glyoxal bis(phenylhydrazone), m. 173-5°. I (5.0 g.) in 20 ml. alc. hydrogenated with 0.5 g. prereduced Pd-C and the filtered solution diluted with 5 ml. H2O, kept at 2-5° and the precipitate, m. 81-6°, recrystd., sublimed and recrystd. gave dihydrooccidentalol (II), m. 86.0-7.8°, [α]25D 65.5° (c 2.3, CHCl3), RRT 1.06, containing less than 2-3% each of tetrahydrooccidentalol (III), RRT 1.15, and epitetrahydrooccidentalol (IV), RRT 0.90. Mixed m.p. of II with eudesmol (V), RRT 1.46 was 72-84°, showing their apparent nonidentity. II (1.0 g.) in 8 ml. MeOH and 2 ml. AcOH hydrogenated slowly with 0.1 g. prereduced PtO2, the filtered solution poured slowly into saturated aqueous NaHCO3 and extracted with Et2O gave material showing 2 peaks of RRT 1.15 (91%) and 0.90 (9%), crystallized from petr. ether, sublimed and recrystd. to yield practically pure III, m. 82.54.0°, [α]25D 42.5° (c 3.4, CHCl3), mixed m.p. with dihydroeudesmol (VI, m. 85.5-6.0°, [α]25D 18.1°, RRT 1.24) 60-82°. I (3.0 g.) in 90% aqueous alc. hydrogenated with 0.3 g. Pd-C and the filtered solution diluted with H2O, extracted with Et2O and the product taken up in 50 ml. Me3COH, stirred intermittently with portionwise alternate addition of 10 g. KMnO4 and 0.5 g. KOH in 200 ml. H2O and the mixture stirred 3 hrs., kept 16 hrs. at 20° and excess KMnO4 destroyed by addition of Na2S2O6, filtered and the filtrate and 90% aqueous Me3COH washings evaporated to a small volume, the crystalline product (0.3 g.) recrystd. from aqueous MeOH and the material, m. 114.5-16.0°, sublimed and recrystd. gave IV, m. 116-17°, [α]25D 9.3° (c 1.6, CHCl3), mixed m.p. 73-88°, with VI. Hydrogenation of I in dry C5H6 gave only 3-5% IV suggesting that 1,4 addition may be involved and that IV is the C-4 epimer of III. If the HOCMe2 group in I and its hydrogenation products is equatorial, then these compds. can differ only from V and VI at the ring juncture and therefore I must have cis-fused rings. Com. V fractionally distilled, the fraction, b10 140-2°, recrystd. repeatedly from petr. ether or MeOH, sublimed, and recrystd. gave a pure isomeric mixture of α- and β-V, m. 81-2°, [α]D 35.0° (c 2.2, CHCl3). V (5.0 g.) in 20 ml. MeOH hydrogenated with 0.25 g. prereduced PtO2, the filtered solution diluted with 3-5 ml. H2O, the solution chilled to 2-5°, and the product recrystd. and sublimed gave VI, m. 85.5-6.0°, [α]25D 18.1° (c 2.16, CHCl3). V (2.0 g.) in 16 ml. MeOH and 4 ml. AcOH hydrogenated 5-6 hrs. at 45-55° with 0.2 g. Pd-C and the filtered diluted solution chilled gave a mixture showing 2 peaks with RRT 0.98 and 1.27 in 53:47 ratio on gas-liquid

chromatography, separated to give epidihydroeudesmol (VII), m. 75.0-82.5° (purified by sublimation and recrystn. to constant m. 73.0-5.5°, [α]D 12.8° (c 1.9, CHCl3), RRT 0.96, and VI, m. 85.0-7.5°, [α]D 15°. Treatment of VI and VII with H and Pd-C 24 hrs. at 20° gave no evidence of epimerization. Assuming equatorial orientation of the HOCMe2 group, VII must be the C-4 epimer and III and IV are the C-4 epimers of VIII. To eliminate the possibility that I and its derivs. are merely C-7 α- epimers of V, the HOCMe2 group was removed by oxidation with CrO3. VI (1.4 g.) in 8 ml. AcOH containing a few drops of H2SO4 and stirred at 80° on a steam bath 1 hr. with dropwise addition of 1.25 g. CrO3 in 24 ml. 1:2 H2O-AcOH, the solution kept 2 hrs. at 80-5° and treated with 1 ml. MeOH, the cooled CrO3-free solution neutralized with NaHCO3 and extracted with Et2O, the product analyzed by gas-liquid chromatography and fractionated on silica gel, eluted with petr. ether to give 0.05 g. hydrocarbon and 0.30 g. 5,9-dimethyl-3-decalone (IX) (semicarbazone m. 219-21°) and finally eluted with Et2O or CHCl3 gave 0.35 g. unreacted VI. III and IV similarly degraded and analyzed gave liquid ketone fractions and starting materials, but the ketone fractions did not show a peak at RRT 0.90, nor was the crystalline semicarbazone, m. 222° obtained. Accordingly since III and IV failed to give IX they must differ from VI at the ring juncture as assumed.

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=> FIL STNGUIDE
COST IN U.S. DOLLARS                          SINCE FILE      TOTAL
                                                   ENTRY    SESSION
FULL ESTIMATED COST                              110.11     110.32

DISCOUNT AMOUNTS (FOR QUALIFYING ACCOUNTS)    SINCE FILE      TOTAL
                                                   ENTRY    SESSION
CA SUBSCRIBER PRICE                              -18.25     -18.25

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FILE CONTAINS CURRENT INFORMATION.
LAST RELOADED: Jan 26, 2007 (20070126/UP).

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YOU HAVE REQUESTED DATA FROM FILE 'CA' - CONTINUE? (Y)/N:Y


L10  ANSWER 9 OF 23  CA  COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER:        108:10995  CA
TITLE:                   Separation of non-polar sesquiterpene olefins from
                         tolu balsam by high-performance liquid
                         chromatography; silver perchlorate
                         impregnation of a prepacked preparative silica
                         gel column
AUTHOR(S):               Friedel, Horst Dieter; Matusch, Rudolf
CORPORATE SOURCE:        Inst. Pharm. Chem. Lebensmittelchem., Philipps-Univ.
                         Marburg, Marburg, 3550, Fed. Rep. Ger.
SOURCE:                  Journal of Chromatography (1987), 407, 343-8
                         CODEN: JOCRAM; ISSN: 0021-9673
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DOCUMENT TYPE: Journal
LANGUAGE: English
AB A separation of nonpolar sesquiterpenoid olefins in EtOH exts. of tolu balsam was achieved by using a AgClO4-loaded HPLC column (LiChrosorb Si 60) and pentane-Et2CO (80:20 and 99:1) as the mobile phase. The method showed high efficiency and good reproducibility, especially in the separation of epimeric compds. which have similar spectroscopic properties.
TI Separation of non-polar sesquiterpene olefins from tolu balsam by high-performance liquid chromatography; silver perchlorate impregnation of a prepacked preparative silica gel column
AB A separation of nonpolar sesquiterpenoid olefins in EtOH exts. of tolu balsam was achieved by using a AgClO4-loaded HPLC column (LiChrosorb Si 60) and pentane-Et2CO (80:20 and 99:1) as the mobile phase. The method showed high efficiency and good reproducibility, especially in the separation of epimeric compds. which have similar spectroscopic properties.
IT Sesquiterpenes and Sesquiterpenoids
RL: BIOL (Biological study)
(olefins, separation from tolu balsam by HPLC of, silver-impregnated silica gel column in)
IT Alkenes, analysis
RL: ANST (Analytical study)
(sesquiterpenoid, separation from tolu balsam by HPLC of, silver-impregnated silica gel column in)
IT Chromatography, column and liquid
(high-performance, stationary phases, silver-impregnated silica gel, for sesquiterpenoid olefins separation in tolu balsam)
IT Balsams
(tolu, sesquiterpenoid olefins separation from, by HPLC, silver-impregnated silica gel column in)
IT 489-39-4 489-40-7 2387-78-2 3856-25-5 22567-17-5 25246-27-9 111778-06-4 111820-84-9 111821-79-5 111900-50-6 111900-51-7
RL: BIOL (Biological study)
(separation of, from tolu balsam by HPLC, silver-impregnated silica gel column in)
IT 7783-93-9, Silver perchlorate
RL: BIOL (Biological study)
(silica gel column impregnated with, for HPLC separation of non-polar sesquiterpenoid olefins)

=> d his

(FILE 'HOME' ENTERED AT 10:23:38 ON 01 FEB 2007)

FILE 'CA' ENTERED AT 10:23:47 ON 01 FEB 2007
L1 89869 S SILICA GEL
L2 4609 S L1 AND LIQUID CHROMATOGRAPH?
L3 2 S UNCOATED AND L2
L4 59244 S REVIEW/TI
L5 3 S UNCOATED SILICA GEL
L6 395 S COATED SILICA GEL
L7 0 S L4 AND L5 AND L6
L8 1 S SEPARAT? EPIMER?
L9 295 S LIQUID CHROMATOGRA? AND EPIMER?
L10 23 S SILICA GEL AND L9
L11 0 S UNCOATED AND L10

FILE 'STNGUIDE' ENTERED AT 10:29:18 ON 01 FEB 2007

10/528179

FILE 'CA' ENTERED AT 10:30:41 ON 01 FEB 2007

FILE 'STNGUIDE' ENTERED AT 10:30:41 ON 01 FEB 2007

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---Logging off of STN---

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Executing the logoff script...

=> LOG Y

COST IN U.S. DOLLARS	SINCE FILE ENTRY	TOTAL SESSION
FULL ESTIMATED COST	0.06	113.96

DISCOUNT AMOUNTS (FOR QUALIFYING ACCOUNTS)	SINCE FILE ENTRY	TOTAL SESSION
CA SUBSCRIBER PRICE	0.00	-18.98

STN INTERNATIONAL LOGOFF AT 10:31:14 ON 01 FEB 2007

CODEN: JOCRAM; ISSN: 0021-9673
DOCUMENT TYPE: Journal
LANGUAGE: English
AB Twenty-six allo bile acids were separated by normal-phase TLC (NP-TLC) and reversed-phase TLC (RP-TLC) as their Me esters; they were also separated by reversed-phase HPLC as their 4-nitrophthalimidemethyl esters. NP-TLC and RP-TLC were on silica gel 60F254 and octadecyl-bonded silica gel RP-18 F2545, resp.; HPLC was on Nova-Pak C18 RP. Results were compared to those for the corresponding 5β epimers.

L10 ANSWER 11 OF 23 CA COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 105:178553 CA
TITLE: Reversed phase high performance liquid chromatographic separation of tetracycline, anhydrotetracycline and their C4-epimers
AUTHOR(S): Iwuagwu, M. A.
CORPORATE SOURCE: Dep. Pharm. Pharm. Technol., Univ. Benin, Benin City, Nigeria
SOURCE: Nigerian Journal of Pharmaceutical Sciences (1986), 2(1), 83-90
CODEN: NJPSEZ; ISSN: 0189-322X
DOCUMENT TYPE: Journal
LANGUAGE: English
AB Tetracycline (I) [60-54-8] and its impurities, epitetracycline [79-85-6], anhydrotetracycline [1665-56-1], and epianhydrotetracycline [7518-17-4] were separated and determined in pharmaceuticals by HPLC. ODS columns bonded to glass beads or silica gel and a mobile phase consisting of MeCN, MeOH and 0.2M phosphate buffer in different ratios were used. The chromatog. was carried out at room temperature, at pH 2.5 with flow rate of 1.5 mL·min-1. The method is simple, rapid, sensitive and suitable as a stability-indicating assay method for study of the kinetics of I degradation The recovery of I was 99.8% with relative standard deviations for I, epitetracycline, anhydrotetracycline and epianhydrotetracycline of 0.11, 0.78, 0.40 and 0.96%, resp.

L10 ANSWER 12 OF 23 CA COPYRIGHT 2007 ACS on STN
ACCESSION NUMBER: 105:746 CA
TITLE: Separation of 7α- and 7β-methoxycarbonylmethyl steroids by preparative high-performance liquid chromatography: comparison with thin-layer chromatography
AUTHOR(S): Charpentier, Bruno; Dingas, Alexandre; Duval, Daniele; Emiliozzi, Romeo
CORPORATE SOURCE: Lab. Chim. Phys. Org., UER Domaine Mediterr., Nice, 06034, Fr.
SOURCE: Journal of Chromatography (1986), 355(2), 427-33
CODEN: JOCRAM; ISSN: 0021-9673
DOCUMENT TYPE: Journal
LANGUAGE: English
AB The 7α- and 7β-carboxymethyl steroid derivs. have been used as heptens in RIA or as ligands in affinity chromatog. TLC separation was performed on activated (110°) silica gel plates with solvents of hexane-EtOAc (30:70), EtOAc, and benzene-(Et)2O (60:40, 30:70) with UV detection. HPLC was performed on μPorasil or reversed-phase μBondapak columns with a mobile phase mixture of hexane-EtOAc-CH2Cl2 of various combinations. Conditions for the separation of each pair of epimers derived from androsterone, cortisone,